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DEVELOPMENT OF A RATING SYSTEM TO DESCRIBE THE FLAWS IN AGED RUBBER SPECIMENS (U)

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DEFENCE RESEARCH ESTABLISHMENT OTTAWA

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ABSTRACT

It was necessary to develop a standardized system of describing the flaws in aged rubber specimens since there seemed to be no correlations in the descriptions by workers in our section. The system described herein has been tested by twenty individuals. Some of these individuals had extensive experience with rubber while others had little or no previous experience.

Using the former method of verbal description there were only two specimens in ten which gave any correlation and both these specimens were devoid of flaws. Utilizing our numerical rating system the percent correlation rose to greater than 70% for the twenty individuals and to over 80% for those workers familiar with the evaluation of flaws in rubber specimens.

RÉSUMÉ

Il fut nécessaire d'établir un système normalisé pour décrire les imperfections dans des échantillons de caoutchouc vieillis, puisqu'il s'est avéré impossible d'obtenir une corrélation avec les descriptions faites par les employés de notre section. Le système présenté ici a été testé par vingt individus. Certains d'entre eux avaient beaucoup d'expérience avec les caoutchoues tandis que d'autres n'avaient aucune expérience préalable.

En employant la méthode de description verbale utilisée précédemment, on n'a pu établir une corrélation que pour deux des dix échantillons qui étaient, par ailleurs, dépourvus d'imperfections. En utilisant le système de gradation numérique le pourcentage de corrélation augmente pour atteindre des valeurs supérieures à 70% pour les vingts individus et à 80% dans le cas des employés familiers avec l'évaluation des imperfections dans le caoutchouc.

EXECUTIVE SUMMARY

Evaluation of the cracking in aged rubber specimens has, in the past, been done very subjectively. The descriptions were wordy and varied from person to person.

Therefore, this system of using a series of measurements and a corresponding number was established and tested. The new numerical system gave greater than 70% correlation whereas with the former method the only correlation that existed was in two specimens that were free from any noticeable flaws.

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1.0 INTRODUCTION

The utility of an objective system for describing flaws generated by the ageing of rubber specimens, is unbounded. As verbal descriptions are bantered around, from lab to lab or even person to person, they become very subjective as the mode of description has never bee standardized.

A thorough search of the literature including International Standards Organization (ISO) recommendations, developed by ISO/TC 45 on rubber and rubber products, as well as, the American Society for Testing and Materials (ASTM Standards) revealed no standardized method for describing the flaws in aged rubber specimens.

It is necessary, within our section to be able to objectively describe the flaws in rubber specimens and accurately convey that description between groups. If a thermal flash experiment on rubber C4 facepieces has taken place, it is necessary to assess the damage on site, since during removal and transportation to DREO the flaws would continue to grow and change. If assessed only on arrival at DREO a true picture of the damage is compromised. Therefore, if everyone within the section uses a standardized method of flaw assessment it can be done on site and subjectivity between groups should be dramatically reduced.

In this report we describe a simple system which we have developed which uses a two digit numbering system, with easy to follow rules, as the method of description. To test this system ten specimens of aged rubber, with differing types of flaws, were given to twenty people for evaluation. The results are reported along with the system description.

2.0 EXPERIMENTAL

2.1 THE RATING SYSTEM

2.1.1 Background

When elastomeric specimens are exposed to the atmosphere, either indoor or outdoor, or in equipment which induces rapid aging, such as an ozone chamber, the final result that one usually observes is a flaw on the surface of the elastomer. The flaw may be large or small (cracks) and there may only be a few, but in some way, this flaw must be described.

Most of the flaws encountered in exposed (aged) elastomeric specimens are due to oxidation by atmospheric oxygen and, especially, ozone. Ozone cracking is characterized by the formation of cracks that are perpendicular to the direction of strain. This causes rapid deterioration of the specimen. Descriptions of these flaws, in the past, have been totally

subjective with little attempt at standardization. Therefore, two people describing the flaws on one specimen would arrive at two different descriptions.

In the literature there are several standards for rubber set by agencies such as the American Society for Testing and Materials (ASTM) and the International Standards Organization (ISO) but none of these standards deal specifically with describing the flaws that occur on the surface of the rubber. ASTM D-518, Rubber Deterioration - Surface Cracking, evaluates comparative performance for resistance to cracking, that is, experiments are conducted to observe and record the time to first crack of a specimen, but there is no scale or system to describe what the flaw looks like. ASTM D-813, Rubber Deterioration - Crack Growth, deals with the determination of crack growth of vulcanized rubber when subjected to repeated bend flexing. Again, there is no provision included to describe the physical appearance of the cracking.

The only standard that includes some sort of system for evaluating the cracking of rubber specimens was ASTM D1171, Rubber Deterioration - Surface Ozone Cracking - Outdoors or Chamber (Triangular Specimens). This standard enables simple comparison of performance of moulded or extruded rubber products through the use of the triangular specimens. The system for describing the cracking is called the "Quality-Retention Rating" and involves establishing a set of values based on three observations of the cracking taken at three successive time intervals of exposure (outdoor or ozone chamber). The specimens are compared to photographs in ASTM D1171 and given an appropriate number at each of the three successive time intervals. A three digit number is then established which corresponds to % Quality Retention of the specimens in question. This method still does not deal with flaw description but rather a projected estimate of properties a specimen will retain as a measure of the rate of crack growth within a specimen over a period of time.

Therefore, it is a necessary requirement, for the groups at DREO, to develop an objective system which will adequately describe the flaws produced in rubber specimens after ageing.

2.1.2 The System

The two most important parameters to define in this system are:

i) The Type of Flaw; and
ii) Percent Coverage/Unit Area.

The type of flaw will be assigned a number. The type of flaw with number assignments are provided in Table 1.

TABLE 1

Type of Flaws	Number
No Flaw	0
Crazing	1
Small Cracks (<3 mm)	2
Medium Cracks (3-7 mm)	3
Large Cracks (>7 mm)	4
Split (completely through)	5

Crazing refers to a surface phenomenon in which the surface of the elastomer looks and feels like alligator skin. Small, medium and large refer to the length of the crack not the depth or the width.

The percent coverage is an assigned number and these are shown below in Table 2.

TABLE 2

Percent Coverage (per unit area, usually 2.5 cm^2)	Number
Zero (0%)	0
Low (1-33%) up to 1/3	1
Moderate (34-50%) 1/3-1/2	2
High (51-100%) > 1/2	3

The system of using photographs was deemod unreliable as the contrast quality was poor. Even in the ASTM-D1171 Standard the quality of the middle two photographs is questionable and therefore, a system which did not rely on photographs, was deemed to be the system of choice for the development of the standard.

2.1.3 **Rating of an Exposed Specimen**

When the exposure period of the specimen is completed, it is examined before* removal from the mounts** and evaluated for the flaws by comparison with the reference standards from Tables 1 and 2. Since the standards in Tables 1 and 2 indicate an area of approximately one square inch (or 2.5 cm^2), it is important to compare the same approximate area from the exposed specimen. The use of a magnifier on the specimen may give better contrast but be sure to measure properly.

2.1.4 **How to Obtain a Two Digit Number**

(A) First examine the specimen and decide if a flaw exists. If not, the specimen gets a 00 rating. If so,

(B) Assign a value of 1 to 5 using the corresponding standards in Table 1.

(C) Now, assign a value of 1 to 3 as shown in Table 2 estimating the percent coverage in the specimen. Remember to designate and explain the location of the area being evaluated.

This system is easy to follow and gives an objective description of flaws in an exposed elastomer specimen.

One problem that may arise, is that more than one type of flaw may be noticeable on a specimen. If so, the major flaw is described first and then the background flaw.

For example, a specimen may show a few large cracks with many medium size cracks. Since the large cracks are the major flaw and there are only a few of them, the rating for this specimen would be 41/33 which says this specimen has a few large cracks with many medium cracks.

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* An examination before removal from the mounts is important because the flaws may change during removal as stress is either removed or applied in the process.

** "Mounts" may refer to wooden or aluminum frames that have the following dimensions: inside width, 100 mm (4 in.); overall width, 175 mm (7 in.); inside length, 300 mm (12 in.); overall length, 380 mm (15 in.): to which rubber strips are mounted: or if the rubber specimen in question is, for example, the entire C4 mask the "mount" could refer to a styrofoam head or a mannequin.

2.2 **THE EXPERIMENT**

To assess the rating system, twenty subjects (some with previous experience) were given 10 exposed* elastomeric specimens and asked to describe flaws, first without the system and then with the system. Each subject was asked to comment on their exercise.

3.0 **RESULTS AND DISCUSSION**

Annex A contains a summary of the verbal descriptions of the ten, aged rubber specimens obtained without reference to the system described above. As expected, these descriptions were subjective and correlations of remarks were non-existent except in specimens B and E where no cracks were visible. To avoid confusion, 6 typical descriptions out of 20 in each case are reported.

In Annex B (Table B-1) the numbering system was used and correlation of results ranged from a low of 50% to a high of 100%, for a non-cracked specimen. The overall reliability of the system is demonstrated by a 73.5% correlation over the ten test specimens. The values for the group of people tested that had experience in assessment of cracking in rubbers showed an 83.75% correlation when the new system of describing the cracking in rubber was used. Table B-2 of Annex B gives the individual results summarized in Table B-1. Table B-2 also shows the results reported by the experienced workers separately.

Some of the comments received indicated that use of a magnifier along with a ruler made the evaluation of the cracking much easier. Also, the area of inspection should be clearly designated. The test specimens were one inch ASTM bent loops, but it is feasible to utilize the standardization procedure on any cracked specimen as long as the examiner clearly defines the location and dimensions of the area to be inspected.

Another comment was that there was no way to describe other variables, such as width or depth or if the crack is jagged or straight. This is true, but the system was devised to describe and standardize what is thought of as routine examinations. If other variables are to be described then these are felt to be fairly specific and may be done with the appropriate adjectives. Width or depth of a crack is very difficult, by any standard, to measure, therefore some subjectivity will still exist.

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* Exposed samples refers to 1 inch ASTM bent loop rubber specimens mounted and exposed outdoors on the roof, according to ASTM-D518. All samples were outdoors 16 months.

4.0 CONCLUSIONS

The rating system presented in this document to describe the flaws in rubber has achieved the goal that was set, that is, much of the subjective description has been eliminated. Some subjectivity still remains but as this system becomes more familiar to those working with it this will be reduced.

ANNEX A

Sample Descriptions
of Cracked Rubber Specimens

SPECIMEN A

Descriptions

Control → Many small cracks
(Author)

widespread minor cracks
tiny multiple cracks
Specimen cracked. High density of cracks about one mm long
Many medium cracks over entire surface
Superficial cracking (<30% of thickness deep)
Cracks over entire upper surface - non-continuous

SPECIMEN B

Control - No cracks

no cracks
no noticeable cracking, possible grayed
no apparent cracking
no cracking or pinholes
no cracks
no visible cracks

SPECIMEN C

Control - Few small cracks

---

few minor cracks near centre of specimen
half a dozen pit point marks
5 small cracks less than ½ mm long
few pinholes, no cracking
sporadic superficial cracks
small cracks ≈ 12 (almost pinholes)

SPECIMEN D

Control - Few small cracks

---

couple of minor cracks
couple of tiny splits 1/10 cm
2 small cracks less than 1 mm long
few small cracks and pinholes
sporadic superficial cracks
pinholes and 2 small cracks

SPECIMEN E

Control - No cracks

---

no visible cracks or pinholes
microscopic edge cracks
no cracking or pinholes
no cracking
no cracking/possibly scuffed
no cracks

SPECIMEN F

Control - Many large cracks

---

specimen partially split, heavy cracking
severe cracking/practically the length of the specimen/
practically through the specimen/wide
severe cracking some up to 2 cm long, specimen almost
split in two
many large cracks covering entire surface
severe deep cracking
continuous cracks over entire surface under stress/
inner material partially revealed

SPECIMEN G

Control - Many large cracks

---

minor surface cracking
severe cracking/small, narrow, parallel, close, straight thin cracks all about 1 cm long
many large deep cracks over entire surface
deep cracking (>60% of thickness)
large continuous cracks - jagged - material almost split

SPECIMEN H

Control - Many large cracks

---

minor surface cracking
rippled cracking, small
many short jagged cracks about 1 cm long
many large to medium cracks, very jagged over entire surface
deep cracking
jagged cracks over entire surface under stress

SPECIMEN I

Control - Many small cracks

---

minor surface cracking
small, diagonal cracking
high density of thin cracks about 2 mm long
many medium cracks over entire surface
superficial cracking
cracks over entire surface

SPECIMEN J

Control - many medium cracks

---

cracks over entire surface
superficial cracking
many medium cracks over entire surface
high density of cracks approx. 2 mm long
small wavy cracks
minor surface cracking

ANNEX B

Descriptions of cracked rubber specimens using Standardized System

Table B-1. Summary of Results Using a Standardized System of Describing Cracking

Specimen	Percent of Correlating Answers	Control	Description
A	50	2,3	Many small cracks
B	100	0,0	No cracks
C	88.3	2,1	Few small cracks
D	88.3	2,1	Few small cracks
E	100	0,0	No cracks
F	67	4,3	Many large cracks
G	67	4,3	Many large cracks
H	67	4,3	Many large cracks
I	50	2,3	Many small cracks
J	67.5	3,3	Many medium cracks
Avg	73.5%		

Table B-2. Individual Results using A Standardized System of Describing Cracking*

SPECIMEN	CONTROL	TOTAL TEST RESULTS	EXPERIENCED WORKER'S RESULTS
A	(2,3)	10 X (2,3), 4 X (3,3), 6 X (2,2)	5 X (2,3) 1 X (3,3)
B	(0,0)	20 X (0,0)	6 X (0,0)
C	(2,1)	17 X (2,1), 3 X (2,2)	5 X (2,1), 1 X (2,2)
D	(2,1)	16 X (2,1), 4 X (2,2)	4 X (2,1), 2 X (2,2)
E	(0,0)	20 X (0,0)	6 X (0,0)
F	(4,3)	14 X (4,3), 5 X (3,3), 1 X (4,2)	5 X (4,3), 1 X (3,3)
G	(4,3)	13 X (4,3), 5 X (3,3), 2 X (4,2)	4 X (4,3), 2 X (3,3)
H	(4,3)	14 X (4,3), 5 X (3,3), 1A X (4,2)	5 X (4,3), 1 X (4,2)
I	(2,3)	10 X (2,3), 5 X (2,2), 5 X (3,3)	5 X (2.3), 1 X (2,2)
J	(3,3)	13 X (3,3), 4 X (3,2), 3 X (2,3)	5 X (3,3), 1 X (2,3)

* These results are summarized in Table B-1.

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It was necessary to develop a standardized system of describing the flaws in aged rubber specimens since there seemed to be no correlations in the descriptions by workers in our section. The system described herein has been tested by twenty individuals. Some of these individuals had extensive experience with rubber while others had little or no previous experience.

Using the former method of verbal description there were only two specimens in ten which gave any correlation and both these specimens were devoid of flaws. Utilizing our numerical rating system the percent correlation rose to greater than 70% for the twenty individuals and to over 80% for those workers familiar with the evaluation of flaws in rubber specimens.

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